# SYNTHESIS AND CHARACTERIZATION OF ELECTRODEPOSITED NICKEL-TUNGSTEN ALLOY THIN FILMS AT DIFFERENT BATH TEMPERATURE

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## ABSTRACT

Electrodeposition technique was used to build Nickel Tungsten composite coatings at varying temperatures in this sample. The effects of temperature on the Ni-W composite coatings' composition, crystal structure, grain size, micro hardness, and surface morphology were analyzed in depth. With increasing temperature, grain size shrank, while micro strain and residual stress increased. At high temperatures, the composite coating's hardness reached its maximum. When the bath temperature was elevated, the tungsten content improved. On the surface, Ni-W films were bright and evenly coated. Ni-W film deposits were also nanoscale, with an average crystalline dimension of 75 nanometers. At 90°C, Ni-W had a micro hardness of 249 VHN.

***Keywords***: *Electrodeposition, electrolytic bath, crystalline size, VSM, Ni-Fe, X-ray diffraction, VHN, SEM*.

## 1. INTRODUCTION

NiW electrodeposits are a good substitute for traditional Ni electrodeposits because of their higher resilience, efficiency, and wear resistance [1-4,7]. These enhanced properties result from tungsten position in microstructure refinement, which reduces the need for organic grain refiners. Since electromagnetically actuated MEMS are more robust for high force and large actuation gap applications, soft magnetic materials are needed for MEMS devices such as micro actuators, sensors, micromotors, and frictionless micro gears. Owing to their use in magnetic storage media, magnetic sensors, and other uses, ferromagnetic films and alloys have special properties relative to non-magnetic films and alloys [6-9]. Because of their low coercivity and strong saturation magnetization, Ni–Fe, Ni–W, and Co–Fe alloys have been widely used. Since the magnetic properties of Ni–W alloys are primarily influenced by their crystal structure, a thorough investigation of this engineering substance is essential for its application areas [10-16]. In recent years, scientists have conducted several experiments on the effects of various deposition parameters on magnetic properties of Ni–W films. Nickel and its alloys have various benefits, including high wear and corrosion resistance [17-20]. As a result, nickel deposition is necessary in the industry to enhance wear and corrosion resistance as well as magnetic properties. Electrochemical methods, such as electrodeposition and electroless deposition, are well-suited to meet the demands of high yield and low cost. The effects of various temperatures on the properties of Ni-W alloy thin films was investigated in this analysis. The preparation and characterization of electroplated Ni-W thin films are described in this article.

## 2. EXPERIMENTAL PART

Temperatures of 30, 50, 70, and 90 º C were used to prepare electroplated Ni-W alloy films. The deposition process took 15 minutes to complete. Copper and stainless steel substrates with dimensions of 1.5 cm x 7.5 cm were used as cathode and anode in this study [21-22]. Electrolytic solution containing sodium tungstate (15 g/l), nickel sulphate (30 g/l), ammonium sulphate (40 g/l), and citric acid (10 g/l) was used to make Ni-W thin films. (22-23). By combining ammonia solution, the pH of the solution was set to 6.0, and the electroplating procedure was carried out at a current density of 2 mA/cm2. After 15 minutes, the copper or cathode was gently removed from the bath and dried for a few minutes [23-24]. Scanning Electron Microscope was used to describe the surface nature of Ni-W films. Energy-dispersive X-ray spectroscopy was used to analyze the mineral composition of film deposits, and X-ray diffraction was used to examine the crystal structure of deposits[25]. Vickers Hardness Test was used to assess the micro hardness of the films.

## 3. RESULTS AND DISCUSSION

### 3.1 COMPOSITION OF Ni-W FILMS

The EDAX results indicate that the films made at higher temperatures contain maximum of tungsten. High temperature yields the highest tungsten content of 24.71 wt percent. The EDAX results indicate that Ni material reduces as temperature rises. Ni-W thin films with low temperatures have a cumulative Ni content of 72.21 wt percent. If the temperature rises, the weight percentage of tungsten increases. The effect of the ammonia solution on the film is neglected since it is only used to fix the pH value of the bath solution.

**Table 1: EDAX analysis of thin films**

| S. No | Temperature (˚C) | Ni Wt% | W Wt% |
|---|---|---|---|
| 1. | 30 | 72.21 | 27.79 |
| 2 | 50 | 65.03 | 34.97 |
| 3 | 70 | 60.42 | 39.58 |
| 4 | 90 | 52.18 | 47.82 |

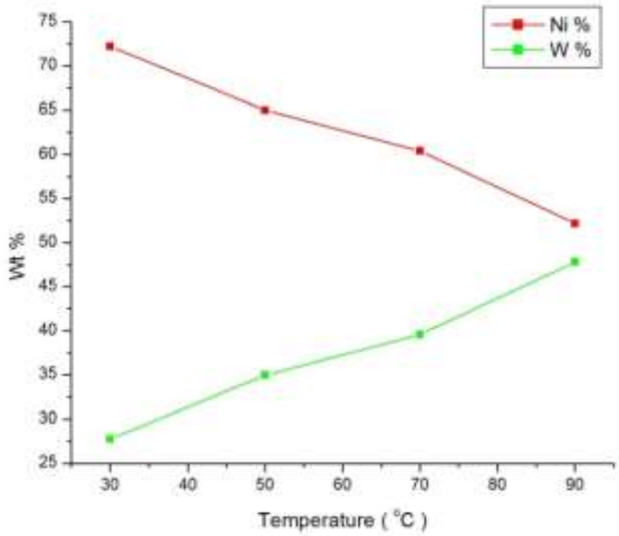
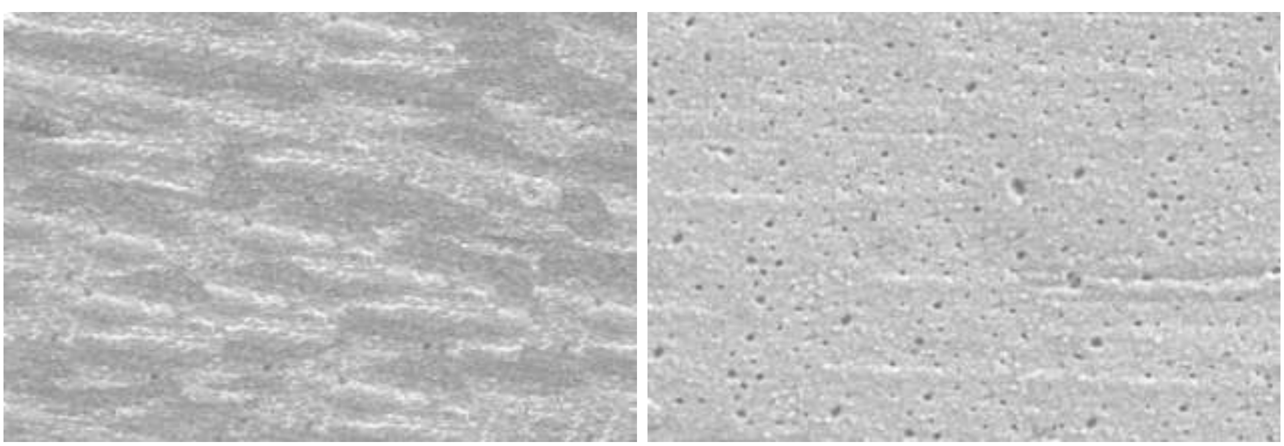


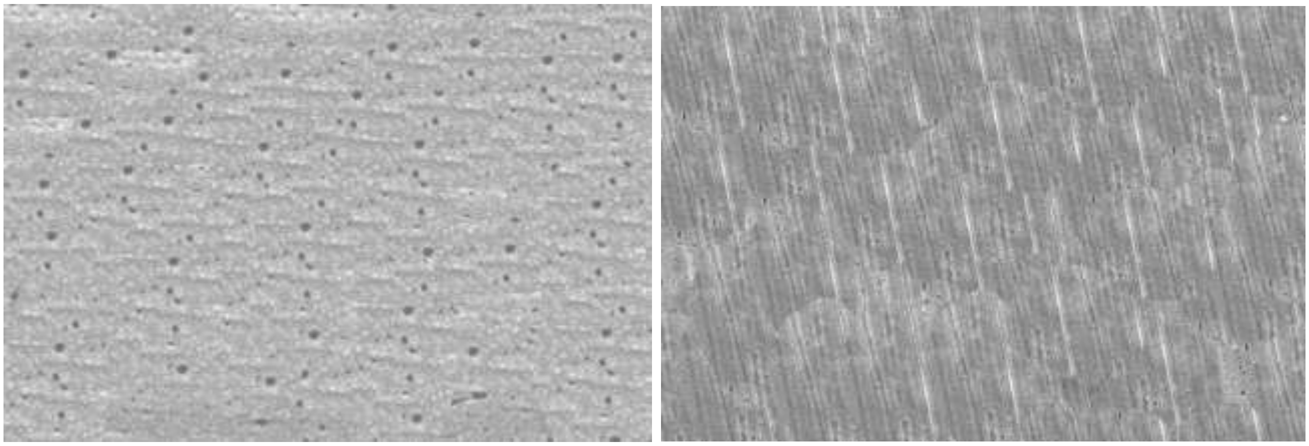

**Figure 1. Variation of nickel and tungsten content with different electrolytic bath temperature**

## 3.2 MORPHOLOGICAL STUDY OF Ni-W FILMS

Scanning Electron Microscope (SEM) images were used to examine the surface structure of Ni-W thin films at temperatures of 30, 50, 70, and 90 ºC, as seen in Fig 2. On the top, the thin films are bright and uniformly coated. They claim to be crack-free.



**(a)** **(b)**



**(c)** **(d)**

**Figure 2.Ni-W films –SEM images at**

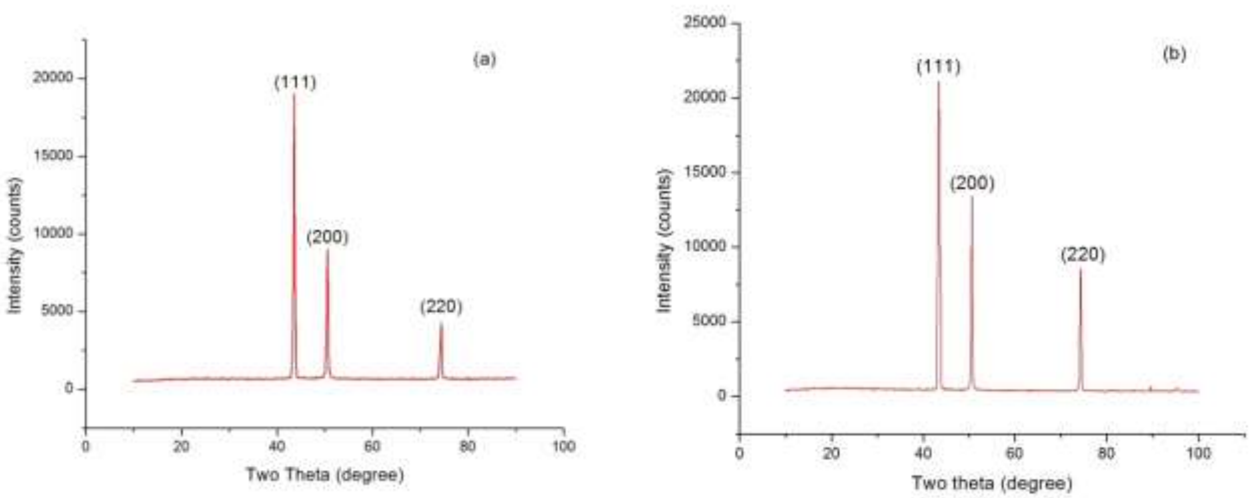
**(a) 30˚C (b) 50˚C (c) 70˚C (d) 90˚C**

### 3.3 STRUCTURAL ANALYSIS OF Ni-W FILMS

XRD processing is used to determine the crystal structure of the electrodeposited Ni-W alloy thin films. Figure 3 shows the X-ray diffraction patterns of Ni-W films obtained at various temperatures. The appearance of sharp peaks in the XRD pattern indicates crystalline existence of the films. XRD is used to measure the crystalline dimension of the deposits using Scherrer's algorithm.

$$D=0.954\lambda/\beta\cos\theta$$

Where $\theta$ is the Bragg's angle , $\lambda$ is the X-ray wavelength, and is the entire range of the diffraction peak at half maximal intensity centered at 2 $\theta$. The XRD patterns of Ni-W films show the presence of the FCC pattern, which has diffraction peaks of (111), (200), and (220). The findings show that the Ni-W deposits' crystalline sizes are obtained by a nanoscale electro deposition technique, with an average crystallite size of about 75 nm.



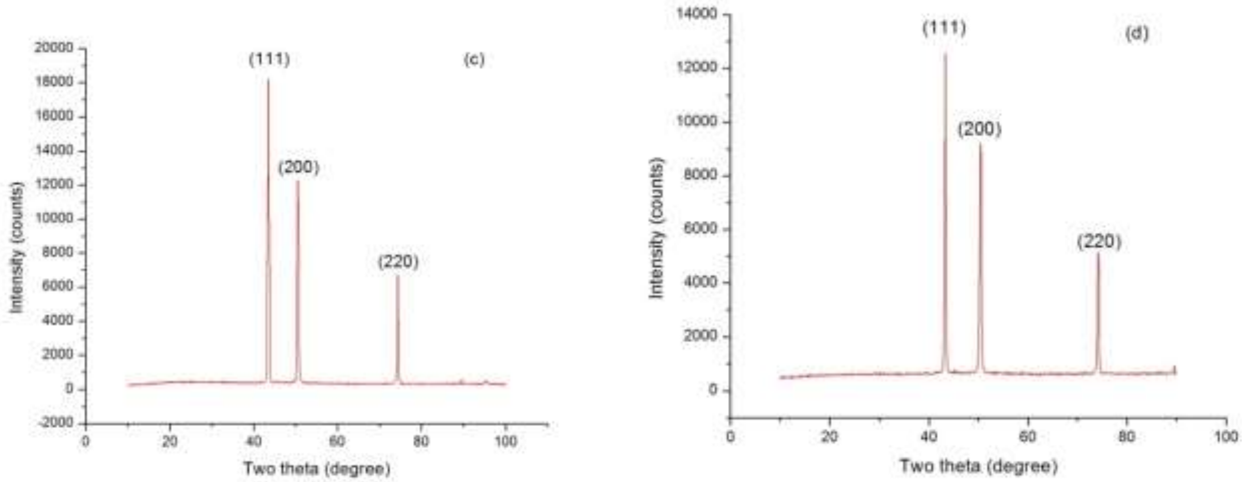



**Figure.3 Ni-W films-XRD patterns at (a) $30^0$C (b) $50^0$C (c) $70^0$C (d) $90^0$C**

The crystal size of Ni-W alloy films is tabulated and shown in table 2. When temperature is increased, the crystalline size of thin films is decreased due to onset orientation of crystals during electrodeposition.

**Table.2: Structural characteristics of Ni-W alloy thin films**

| S.No | Bath Temperature (˚C) | 2θ (deg) | d ($A^0$) | Particle Size(D) (nm) |
|---|---|---|---|---|
| 1 | 30 | 43.47 | 1.6012 | 95.67 |
| 2 | 50 | 44.93 | 1.4914 | 75.04 |
| 3 | 70 | 43.05 | 1.3814 | 70.42 |
| 4 | 90 | 42.93 | 1.3017 | 59.53 |

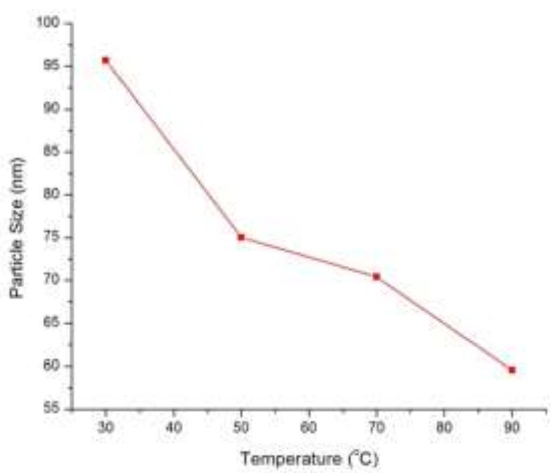



**Figure 4. Variation of crystal size with different electrolytic bath temperature**

### 3.4 MECHANICAL PROPERTIESOF Ni-W FILMS

Vickers hardness tester was used to measure the micro hardness of the deposits. Thin films prepared at temperatures of 30, 50, 70, and 90 $^{o}$C have hardness values of 124,156,182, and 249 VHN, respectively. As a result of the lower tension associated with thin films, micro hardness increases as the electrolytic bath temperature rises. Figure 5 illustrates how hardness changes as bath temperature rises.

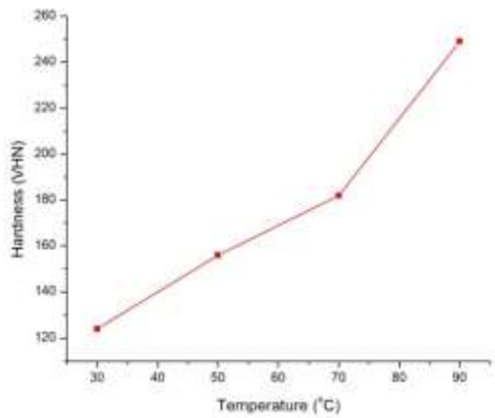



**Figure 5. Variation of micro hardness with different electrolytic bath temperature**

## 4. CONCLUSION

Ni-W alloy thin films have been successfully synthesized using electrodeposition at varying temperatures. Crack-free, bright, and uniform nano crystalline films are obtained at various temperatures. Electrodeposited Ni-W thin films have a dominant composition of FCC. The deposits have crystalline sizes in the nanometer range. When the temperature rises, so does the hardness. The particle size values fall from 95.67 nm to 59.53 nm as the temperature rises from 30 to 90 degrees Celsius. This occurs as a result of Ni-nanocrystalline W's composition and low film stress. This article outlines the optimum electroplated bath operating conditions. Ni-W thin films can be used in a number of electronic instruments, such as high-density storage media, magnetic writing heads, high-performance transformer cores and MEMS.

## REFERENCES

1. Nosang,V.; Park,D.Y.; Yoob,B.Y.; Paulo,T.A.; Development of electroplated magnetic materials for MEMS, Journal of Magnetism and Magnetic Materials, 2003, 265 , 189-198.
2. Osaka, T.; A soft magnetic CoNiFe film with high saturation magnetic flux density and low coercivity, Nature, 1998, 392, 796 – 798.
3. B. Tury, M. Lakatos-Varsányi, S. Roy, Ni–Co alloys plated by pulse currents, Surf.Coat. Technol. 200 (2006) 6713–6717.
4. Iwasaki,S.;Nakamura,Y.;An analysis for the magnetization mode for high density magnetic recording, Journal of Magnetism and Magnetic Materials, 1977, 200, 634-648.

5. Esmaili, S.; Bahrololoom, M.E.; Zamani, C.; Electrodeposition of NiFe/Cu multilayers from a single bath, Electron. Process. Mater., 2011, 47(2) 10-15.
6. L. Chang, C.-H. Chen, H. Fang, Electrodeposition of Ni–P alloys from a sulfa-mate electrolyte relationship between bath pH and structural characteristics,J. Electrochem. Soc. 155 (2008) D57.
7. L. Shi, C.F. Sun, P. Gao, F. Zhou, W.M. Liu, Electrodeposition and characterizationof Ni–Co–carbon nanotubes composite coatings, Surf. Coat. Technol. 200 (2006)4870–4875.
8.Myung,N.; A Study on the Electrodepositionof NiFe Alloy Thin Films Using Chronocoulometry andElectrochemical Quartz Crystal Microgravimetry, Bull. Korean Chem. Soc.,2001,22, 994-998.
9. M. Hakamada, Y. Nakamoto, H. Matsumoto, H. Iwasaki, Y.Q. Chen, H. Kusuda,M. Mabuchi, Relationship between hardness and grain size in electrodepositedcopper films, Mat. Sci. Eng. A 457 (2007) 120.
10. E. Gómez, J. Ramirez, E. Valles, Electrodeposition of Co–Ni alloys, J. Appl. Elec-trochem. 28 (1998) 71–79.
11. B. Bakhit, A. Akbari, Synthesis and characterization of Ni–Co/SiC nanocom-positecoatings using sediment co-deposition technique, J. Alloys Compd. 560(2013) 92–104.
12. Motomura, Y. ; Tatsumi, T.; Urai, H.; Aoyama, M.; Soft magnetic properties and heat stability for Fe/NiFe superlattices, IEEE Trans. Magn., 26 ,1990, 2327-2331.
13. Wang,S.L.; Studies of electroless plating of Ni-Fe-P alloys and the influences of some depositionparameters on the properties of the deposits, Surface and Coatings Technology,2004,186, 372–376.
14. Dixit, G.; Singh, J.P.; Srivastava, R.C. ; Agrawal, H.M.; Choudhary, R.J.; Ajay, G.; Structural and magnetic behaviour of NiFe2O4 thin film grown by pulsed laser deposition, Indian J. Pure Appl. Phys., 2010,48 , 287-291.
15.Hamid, Z.A.; Electrodeposition of Cobalt- Tungsten Alloys from Acidic Bath Containing Cationic Surfactants, Materials Letters,2003,57, 2558.
16.An,Z.G.; Zhang,J.J.; Pan,S.L.; Fabrication of glass/Ni-Fe-P ternary alloy core/shell composite hollow Microspheresthrough a modified electroless plating process, Applied Surface Science,2008, 255, 2219–2224.
17. G. Qiao, T. Jing, N. Wang, Y. Gao, X. Zhao, J. Zhou, W. Wang, High-speed jet electrodeposition and microstructure of nanocrystalline Ni–Co alloys, Electrochim.Acta 51 (2005) 85–92.
18. C.V. Thompson, Structure evolution during processing of polycrystalline films,Annu. Rev. Mater. Sci. 30 (2000) 159.
19. Meenu Srivastava, V. Ezhil Selvi, V.K. William Grips, K.S. Rajam, Corrosion resis-tance and microstructure of electrodeposited nickel–cobalt alloy coatings, Surf.Coat. Technol. 201 (2006) 3051–3060.
20. B. Ranjith, G. Paruthimal Kalaignan, Ni–Co–TiO2nanocomposite coating pre-pared by pulse and pulse reversal methods using acetate bath, Appl. Surf. Sci.257 (2010) 42–47.
21. Fernandez,G.V; Grundy,P.J.; Vopson,M.M.; Control and analysis of grain Size in sputtered NiFe thin films, J. Phys, Condens. Matter,2013, 1(1),6-9.
22. Kuru, H.; Kockar, ; Alper, M.; Karaagac, O.; Growth of binary Ni–Fe films: Characterisations at low and high potential levels, J. Magn. Magn. Mater., 2015, 377 , 59-64.
23.Esther, P.; Joseph Kennady,C.; Effect of sodium tungstate on the properties of Electrodeposited nanocrystalline NiCoCr films,Journal of Non Oxide Glasses., 2010, 1, 35-44.
24. B. Bakhit, A. Akbari, Nanocrystalline Ni–Co alloy coatings: electrodepositionus-ing horizontal electrodes and corrosion resistance, J. Coat. Technol. Res. 10 (2)(2013) 285–295.
25. Fernandez ,G.V; Grundy, P.J.; Vopson, M.M.; Control and analysis of grain Size in sputtered NiFe thin films, J. Phys, Condens. Matter, 2013, 1(1),6-9.